

CLAIMS

- 1 A micro-engineered reactor for performing chemical synthesis, said reactor comprising
:
5 (a) a reaction chamber and an electrochemical cell in close communication with the
interior of the reaction chamber;
 (b) said reaction chamber being of micro-engineered dimensions and having micro-
engineered inlet and outlet conduits for conveying fluids to, and from, the reaction
chamber;
10 (c) said cell comprising at least two electrodes positioned and arranged relative to
each other to define a space between them, which, in at least one mode of use of
the cell, receives an ionically conductive fluid that is, or contains, a redox-active
source material, and forms an electrically conductive path between the electrodes
through which an electrical current passes when an electrical potential is applied to
15 the electrodes so as to electrolyse the source material and form a primary product
at, or adjacent to, a surface of an electrode within the interior of the reaction
chamber;
 (d) means for introducing into the reaction chamber a precursor material that will react
with the primary product to produce a secondary product, and
20 (e) means for conveying the secondary product away from the reaction chamber
through said outlet conduit.
- 2 A reactor according to claim 1 wherein at least one of the electrodes has a surface at
which, in use, the primary product is formed, and the surface is located within, or forms
25 a part of a boundary wall of, the reaction chamber.

- 3 A reactor according to claim 1 wherein at least one of the electrodes has a surface at which, in use, the primary product is formed, made of, or coated with, a sacrificial material that will react with the source material to form the primary product,
- 5 4 A reactor according to claim 3 wherein the cell further includes an electro-depositing means for selectively applying an electrical charge to each of the electrodes in the presence of an ionically conductive fluid that contains ions of the sacrificial material, thereby to cause the sacrificial material to be deposited on the electrode or electrodes and replenish the sacrificial material that is consumed by the electrolysis.
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- 5 A reactor according to claim 4 wherein the electro-depositing means is operable to deposit the sacrificial material on the surface of the respective electrode or electrodes prior to, or during reaction of the sacrificial material with the source material to form the primary product.
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- 6 A reactor according to claim 4 wherein the electro-depositing means is operable to deposit the sacrificial material on the surface of the respective electrode or electrodes prior to, or during reaction of the sacrificial material with the source material to form the primary product.
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- 7 A reactor according to claim 4 wherein the electro-depositing means comprises means for applying a negative charge to one of the electrodes and a positive charge to the other electrode, and a fluid supply means is provided for introducing a source of an organic fluid containing a salt in which a cation is the sacrificial material to be deposited
- 25 on the negatively charged electrode, and a means is provided for generating an electrical field between the two electrodes so as to cause electro-deposition of the material on to the surface of the negatively charged electrode.

- 8 A reactor according to claim 4 wherein the electro-depositing means comprises means
for applying a negative charge to one of the electrodes and a positive charge to the
other electrode, and a fluid supply means is provided for introducing a source of an
5 organic fluid containing a salt in which an anion is the sacrificial material to be
deposited on the positively charged electrode, and a means is provided for generating
an electrical field between the two electrodes so as to cause electro-deposition of the
material on to the surface of the positively charged electrode.
- 10 9 A reactor according to claim 7 wherein the organic fluid that contains the ions of the
sacrificial material is also the organic reagent that will react with the deposited material
to produce the primary product.
- 10 A reactor according to claim 7 wherein the organic fluid that contains the ions of the
15 sacrificial material is also the precursor that will react with the deposited material to
produce the secondary product.
- 11 A reactor according to claim 1 wherein at least one of the electrodes is made of a fluid
permeable material and defines a part of the boundary wall of the reaction chamber
20 through which a fluid source material permeates to react at a surface of the fluid
permeable electrode that is in communication with the interior of the reaction chamber
to form the primary product.
- 12 A reactor according to claim 11 wherein a second chamber is formed on a side of the
25 fluid permeable electrode that is remote from the interior of the reaction chamber, and
an electrode of the cell is located in the second chamber, said fluid permeable
electrode being made of a material that allows a primary product created by electrolysis

of a source material within the second chamber to permeate through the permeable electrode into the interior of the reaction chamber and to react with the precursor material to form the secondary product.

- 5 13 A reactor according to claim 11 wherein the second chamber has an inlet conduit for the supply of fluid containing fresh source material to the second chamber and an outlet for removal from the second chamber of fluids depleted of said source material.
- 10 14 A reactor according to claim 11 wherein a fluid permeable ionic or electronic conductor is provided between the electrodes so as to define a first region on one side of the conductor where an ion conductive solution is retained in contact with the one of the electrodes, and a second region on the other side of the said conductor, where the conductor is in contact with the source material.
- 15 15 A reactor according to claim 14 wherein the conductor is mercury or an amalgam.
- 16 A reactor according to claim 1 wherein the cell is a rechargeable cartridge.
- 17 A reactor according to claim 1 wherein there is provided a fluid pumping means
20 connected to the reactor for pumping the fluids through the reactor.
- 18 A reactor according to claim 1 wherein the source material and the precursor are present in a common fluid.
- 25 19 A reactor according to claim 1 wherein the source material is present in a first fluid and the precursor is present in a second fluid.

- 20 A reactor according to claim 1 wherein the outlet conduit of the reaction chamber is divided into two or more discrete outlet paths.
- 21 A reactor according to claim 20 wherein at least one or more discrete outlet paths are
5 connected to an inlet conduit of another reactor constructed in accordance with claim 1.
- 22 A reactor according to claim 20 wherein one of the electrodes of the cell is located in, or forms part of, the wall of the reaction chamber and one of the electrodes of the cell is located in or forms part of the wall of a conduit defining one of the discrete outlet paths.
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- 23 A reactor according to claim 1 wherein the reaction chamber is constructed, dimensioned and arranged to define a plurality of adjacent flow paths through the reaction chamber via respective pairs of an inlet conduit and an outlet conduit.
- 15 24 A reactor according to claim 1 wherein there is provided an ion permeable barrier located between an anode and a cathode of the cell so as to define an anodic compartment adjacent the anode on one side of the ion permeable barrier and a cathodic compartment adjacent the cathode on the other side of the ion permeable barrier.
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- 25 A reactor according to claim 24 wherein the barrier is selected from the following materials, namely a porous or permeable sheet, a dialysis membrane, an ion exchange membrane, a region of gelled electrolyte or a region of immobilised electrolyte.
- 25 26 A reactor according to claim 1 wherein at least one of the electrodes is a porous block through which fluids may flow.

- 27 A reactor according to claim 1 wherein there is provided a first discrete inlet conduit for supplying the source material, a second discrete inlet conduit for supplying the precursor, and a third discrete conduit for supplying a flushing fluid for removing the secondary product from the reaction chamber.
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- 28 A reactor according to claim 27 wherein valve means are provided for selectively opening and closing each of the first, second or third inlet conduits.
- 29 A reactor according to claim 1 wherein one or more reference electrodes means is provided in the reaction chamber or cell for monitoring and controlling the electrical potential applied to the electrodes of the cell.
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- 30 A reactor according to claim 29 wherein one or more monitoring electrode means is provided for monitoring the composition of the products produced in the reactor.
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- 31 A reactor according to claim 1 wherein there is provided within, or adjacent to, the reaction chamber, a heat exchanger for controlling the temperature of at least a region of the interior of the reaction chamber.
- 20 32 A reactor according to claim 1 wherein the output conduit is connected in flow series to an inlet conduit of another reactor constructed in accordance with claim 1.
- 33 A reactor according to claim 32 wherein a plurality of series connected reactors are provided on a common structure.

- 34 A reactor according to claim 32 wherein one or more arrays of series connected
reactors are arranged relative to another one or more arrays of series connected
reactors on a common structure.
- 5 35 A method of performing chemical synthesis comprising the steps of:
- (a) providing a reactor constructed in accordance with claim 1
- (b) presenting a source material to the space between at least two electrodes;
- (c) electrolysing the source material to form a primary product at, or adjacent to, a
10 surface of the electrode that communicates with the interior of the reaction
 chamber; and,
- (d) contacting the primary product in the reaction chamber with a precursor that
 will react and/or mix with the primary product to produce a secondary product.
- 15 36 A method according to claim 35 including the step of removing the secondary product
 from the reaction chamber.
- 37 A method according to claim 36 including the step of reacting the secondary product
 with a material that is a precursor for use in a further reaction, or a series of further
20 reactions, to produce a final product.
- 38 A method for preparing a synthetic organic compound comprising the steps of:
- (a) providing a micro-engineered reactor constructed in accordance with claim 1
 wherein at least one of the electrodes is made of, or coated with, a sacrificial
25 material that will react with the precursor to form the secondary product;
- (b) presenting a source material to at least the electrode that consists of, or has
 deposited thereon, the sacrificial material;

- (c) electrolysing the source material and allowing the electrolysed source material to react with the sacrificial material to form the primary product at, or adjacent to a surface of the electrode; and,
- (d) contacting the primary product in the reaction chamber with a precursor that will react with the primary product to form the said organic compound.

39 A method for preparing a synthetic organic compound comprising the steps of:

- (a) providing a micro-engineered reactor constructed in accordance with claim 1 wherein at least one of the electrodes is made of, or coated with, a sacrificial material that will react with the precursor to form the secondary product;
- (b) presenting an organic source material to at least the electrode that consists of, or has deposited thereon, the sacrificial product;
- (c) electrolysing the source material and allowing the source material to react with the sacrificial material to form the secondary product at, or adjacent to a surface of the electrode; and,
- (d) contacting the primary product in the reaction chamber with a precursor that will react with the primary product to form the said organic compound.

40. A method according to claim 38 further including the step of replenishing the consumed sacrificial material by electro depositing material on at least one of the electrodes.

41. A method according to claim 40 wherein the sacrificial material is deposited on the respective electrode or electrodes by connecting the electrodes to an electrical supply whereby one of the electrodes forms a cathode and the other forms an anode, and the method includes the steps of presenting to the cathode, a fluid containing a salt in which a cation is the sacrificial material, and applying an electrical field between the

cathode and an anode thereby to deposit the sacrificial material on the cathode.

- 5 42. A method according to claim 40 wherein the sacrificial material is deposited on a surface of the respective electrode by connecting the substrate to an electrical supply whereby one of the electrodes forms a cathode and the other forms an anode, and the method includes the steps of presenting to the anode, an ionically conductive fluid containing a salt in which an anion is the sacrificial material, and applying an electrical field between the cathode and an anode thereby to deposit the sacrificial material on the anode.
- 10 43. A method according to claim 41 wherein a fluid containing the salt is presented to the electrodes whilst simultaneously applying an electrical charge to the electrodes thereby to deposit the sacrificial material onto at least one electrode, and the source material is subsequently presented to the electrodes and allowed to react electrolytically with the sacrificial material to form the primary product
- 15 44. A method according to claim 41 wherein a fluid containing the salt is presented to the electrodes whilst simultaneously applying an electrical charge to the cathode thereby to deposit the sacrificial material onto at least one electrode, and the source material is presented to the electrodes simultaneously with the salt, and allowed to react with the deposited sacrificial material to form the primary product.
- 20 45. A method according to claim 35 wherein the reactor is constructed in accordance with claim 12 and said method includes steps of:
- 25 (a) introducing into the second chamber a fluid containing said source material; and

(b) electrolysing said source material in said second chamber so as to produce said primary product at a surface of the fluid permeable electrode that is in communication with the interior of the reaction chamber.

5 46. A method according to claim 35 for the synthesis of lithium, or sodium, or potassium alkyls wherein the source material is derived from solutions of lithium, or sodium, or potassium salts, or solid or polymeric ion conductors thereof, and the precursor is an alkyl halide that reacts with the source material to form lithium, or sodium, or potassium alkyls as the secondary product.

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47. A method according to claim 35 for the synthesis of Sodium Naphthalenide wherein the source material is sodium, the precursor is naphthalene and the secondary product is sodium Naphthalenide.

15 48. A method according to claim 35 for the synthesis of potassium Naphthalenide wherein the source material is potassium, the precursor is naphthalene and the secondary product is potassium Naphthalenide.

49. A method according to claim 35 for the synthesis of Sodium anthracenide wherein the
20 source material is sodium, the precursor is anthracene and the secondary product is sodium anthracenide.

50. A method according to claim 35 for the synthesis of potassium anthracenide wherein
25 the source material is potassium, the precursor is anthracene and the secondary product is potassium anthracenide

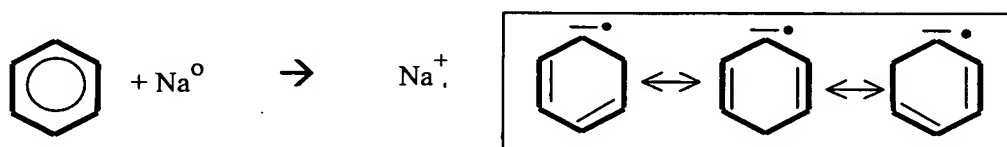
51. A method according to claim 35 for the synthesis of Sodium phenanthrenide wherein the source material is sodium, the precursor is phenanthrene and the secondary product is sodium phenanthrenide
- 5 52. A method according to claim 35 for the synthesis of Potassium phenanthrenide wherein the source material is potassium, the precursor is phenanthrene and the secondary product is potassium phenanthrenide
53. A method according to 35 for the conversion of organotin compounds wherein the
10 source material is sodium, the precursor is an organotin compound.
54. A method according to claim 53 for carrying out the conversion of organotin compounds by a reaction of the type
- $$\text{Na}^+ + \text{e}^- (\text{from electrode}) \rightarrow \text{Na}^0$$
- 15
$$(\text{CH}_3)_3\text{Sn}^- \text{Sn}(\text{CH}_3)_3 + 2 \text{Na}^0 \rightarrow 2 (\text{CH}_3)_3\text{Sn}^- \text{Na}^+$$
55. A method according to claim 54 wherein the sodium compound is further reacted with an organo halide in accordance with the following formula:
- $$(\text{CH}_3)_3\text{Sn}^- \text{Na}^+ + \text{CH}_2=\text{CH}-\text{CH}-\text{Cl} \rightarrow (\text{CH}_3)_3\text{Sn}-\text{CH}_2.\text{CH}=\text{CH} + \text{Na}^+ \text{Cl}^-$$
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- 56 A method according to claim 35 for the generation of sodium, lithium or potassium alkoxide or phenoxide in accordance with formula
- $$\text{Na/K/Li}^+ + \text{e}^- (\text{from electrode}) \rightarrow \text{Na/K/Li}^0$$
- $$\text{Na/K/Li}^0 + \text{R}^-\text{OH} \rightarrow \text{Na/K/Li}^+ \text{OR}^- + \text{H}_2$$
- 25 where R = CH₃ , C₂H₅, C₃H₇, C₄H₉, Ph, substituted Ph, heteroaryl, or substituted heteroaryl

- 57 A method according to claim 35 for the removal of halides from aromatics by sodium metal in accordance with the reaction



where Hal = Cl, Br, or I

- 58 A method according to claim 35 for carrying out a Birch Reduction of compounds in accordance with the reaction:



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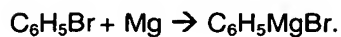
- 59 A method according to claim 35 for the generation of Grignard reagents in accordance with the reaction:



where R = C₆H₅, and X = Cl, Br, or I

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- 60 A method according to claim 59 wherein the reaction is:



- 61 A method according to claim 35 for the generation of halogenated products wherein the source material is a soluble halide, the primary product is a halogen produced by the electrochemical oxidation of the soluble halide, and the halogen is reacted with an

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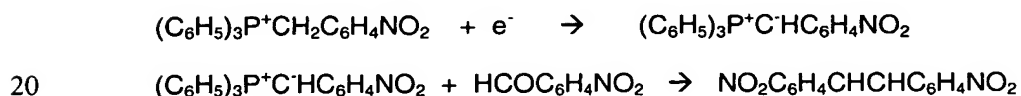
organic precursor to produce a secondary halogenated product by addition to unsaturated bonds or by substitution.

62 A method according to claim 35 for the generation of halogenated products wherein the
5 source material is a soluble halide, the primary product is a halogen produced by the electrochemical oxidation of the soluble halide, and the halogen is reacted with an inorganic precursor to produce an inorganic reactive intermediate.

63 A method according to claim 61 wherein the halogen is chlorine, the inorganic
10 precursor is phosphorus, and the secondary product is PCl_3 or PCl_5 .

64 A method according to claim 61 wherein the secondary product is reacted in the reaction chamber with a carboxylic acid to generate acyl chloride products.

15 65 A method according to claim 35 for the generation of ylids from phosphonium salt cations by the Wittig reaction and the reaction of ylids and aldehyde to generate alkenes in accordance with the formulae :



66 A reactor according to claim 8 wherein the organic fluid that contains the ions of the sacrificial material is also the organic reagent that will react with the deposited material to produce the primary product.

- 67 A reactor according to claim 8 wherein the organic fluid that contains the ions of the sacrificial material is also the precursor that will react with the deposited material to produce the secondary product.
- 5 68 A reactor according to claim 21 wherein one of the electrodes of the cell is located in, or forms part of, the wall of the reaction chamber and one of the electrodes of the cell is located in or forms part of the wall of a conduit defining one of the discrete outlet paths.
- 69 A method according to claim 39 further including the step of replenishing the consumed
10 sacrificial material by electro depositing material on at least one of the electrodes.
- 70 A method according to claim 42 wherein a fluid containing the salt is presented to the electrodes whilst simultaneously applying an electrical charge to the electrodes thereby to deposit the sacrificial material onto at least one electrode, and the source material is
15 subsequently presented to the electrodes and allowed to react electrolytically with the sacrificial material to form the primary product.
- 71 A method according to claim 42 wherein a fluid containing the salt is presented to the electrodes whilst simultaneously applying an electrical charge to the cathode thereby to
20 deposit the sacrificial material onto at least one electrode, and the source material is presented to the electrodes simultaneously with the salt, and allowed to react with the deposited sacrificial material to form the primary product.
- 72 A method according to claim 62 wherein the halogen is chlorine, the inorganic
25 precursor is phosphorus, and the secondary product is PCl_3 or PCl_5 .

- 73 A method according to claim 62 wherein the secondary product is reacted in the reaction chamber with a carboxylic acid to generate acyl chloride products.